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INDIANA DEPARTMENT OF TRANSPORTATION
MATERIALS AND TESTS DIVISION

# DETERMINATION OF LIME CONTENT IN SOIL
ITM No. 503-96T

## 1. SCOPE

1.1 This method describes a procedure to determine the lime content of treated soils by flame atomic absorption spectrophotometry. The determination is accomplished by analysis of elemental calcium in a diluted, acid-digested solution of the sample material, and mathematical conversion of the calcium value to expression as lime content, corrected for calcium background in untreated soil.

1.2 This test method involves hazardous materials, operations, and equipment. This method does not purport to address all of the health or safety problems associated with its use. It is the responsibility of whoever uses this method to consult and establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

## 2. REFERENCED DOCUMENTS

2.1 AASHTO Standards
T 232 Determination of Lime Content in Lime-Treated Soils by Titration
M 231 Specifications for Weighing Devices Used in the Testing of Materials

2.2 ASTM Standards
C 25 Chemical Analysis of Limestone, Quicklime, and Hydrated Lime
D 1193 Specification for Reagent Water
E 663 Practice for Flame Atomic Absorption Analysis
E 694 Specification for Volumetric Ware
E 832 Specification for Laboratory Filter Paper

2.3 Other Standards
American Chemical Society Specifications, Reagent Chemicals
US EPA SW-846 Test Method 7000

## 3. SIGNIFICANCE AND USE

3.1 This test method as described in Section 1.1 is a laboratory test method. It was developed as an alternate to AASHTO T 232, which is a field method that is limited due to the specified time restrictions between the addition of lime and the analysis of the treated soil.

3.2 This test method requires that the lime used for treatment of the soil shall be analyzed for calcium in accordance with ASTM C 25. It also requires that for each sample of treated soil that is analyzed shall have a background sample of soil analyzed. Both the background sample and treated soil sample shall be taken at the same depth and location.

3.3 Soils which contain a high content of aluminum, sulfates or phosphates may cause chemical or spectral matrix interferences which can cause erroneous results. The use of lanthanum reduces these interferences, but some types of soil may require further extraction.

4. APPARATUS

4.1 Flame Atomic Absorption Spectrophotometer (Note 1)

4.2 Chemical Fume Hood with Electric Hot Plate

4.3 Analytical Balance, AASHTO M 231, Class A

4.4 Volumetric Flask, ASTM E 694, Class A (Note 2)

4.5 Filter Paper, ASTM E 832, Type II, Class F

4.6 Miscellaneous Laboratory Glassware (Note 2)

5. REAGENTS

5.1 Nitric acid, concentrated, specific gravity 1.42, ACS reagent grade

5.2 Reagent Water, ASTM D 1193, Type II

5.3 Calcium Standard Solution, 1000 µg/mL Ca, prepared from ACS primary grade calcium carbonate previously dried 2 hours at 285 °C

5.4 Lanthanum Solution, 25 000 µg/mL La, prepared from ACS reagent grade lanthanum chloride or lanthanum oxide

6. SAMPLE PREPARATION

6.1 Weigh separately into 250 mL griffin beakers 1.0000 ± 0.0005 grams of the dried soil blank and of the dried lime treated soil. (Note 3)

6.2 In a chemical fume hood, add 20 mL of reagent water and 10 mL of nitric acid, stir to break up any lumps of soil with a glass stirring rod and cover with a watch glass.

6.3 After the effervescence subsides, dilute the solution to 50 mL with hot reagent water and heat to a boil. Digest the solution covered at a temperature just below boiling for one hour.

6.4 Filter the hot solution into a 250 mL volumetric flask, washing the beaker and filter paper with a hot reagent water, cool and dilute to volume with reagent water and mix.

7. CALCIUM ANALYSIS

7.1 Prepare instrument calibration standards at concentrations of 0, 1, 2.5 and 4 µg/mL calcium with each containing 1000 µg/mL lanthanum.

7.2 From the solutions prepared in 6.4 take aliquots into volumetric flasks which will provide concentrations of 1 to 4 µg/mL calcium, add an aliquot of lanthanum solution that will provide 1000 µg/mL lanthanum, dilute with reagent grade water to volume and mix.

7.3 Calibrate the spectrophotometer with the calibration standards in accordance with ASTM E 663 and the instrument manufacturer's recommendations using a wavelength of 422.7 nanometers, slit width of 0.2 nanometer, acetylene-nitrous oxide flame and background correction.

7.4 Aspirate the diluted soil blank and limed soil solutions prepared in 7.2 and determine the calcium concentrations. (Note 4)

8. CALCULATIONS

8.1 Calcium % by Mass

$$\text{Calcium by Mass \%} = \frac{A \times V \times D \times K}{W}$$

where:

A = µg/mL of calcium determined
V = 250 mL, volume of volumetric flask in 6.4
D = Dilution factor in 7.2 = $\frac{\text{volume of final solution}}{\text{volume of aliquot}}$
K = $1 \times 10^{-4}$
W = Mass of sample in grams

8.2 Lime % by Mass of Dried Treated Soil

$$\text{Lime \% by Mass} = \frac{CT - CB}{CL} \times 100,$$

where:

CT = % of calcium by mass of treated soil
CB = % of calcium by mass of soil blank
CL = % of calcium by mass of lime used for treating soil

9. PRECISION AND BIAS

9.1 No study has been undertaken to determine the precision or bias of this test method due to the high degree of variations in soil types, sampling, and matrix interferences.

10. NOTES

10.1 A Varian-Techtron model AA-6D spectrophotometer was used in the development of this test method. Differences between the various makes and models of satisfactory atomic absorption spectrophotometers prevent the formulation of detailed instructions or specifications applicable to every instrument. As a minimum the spectrophotometer shall have a detection limit of 0.002 µg/mL, sensitivity of 0.013 µg/mL and an optimum working range of one to 4 µg/mL for the determination of calcium.

10.2 All glassware used for this test method must be cleaned in the following sequence to prevent contamination: detergent; tap water; 1:1 nitric acid; tap water; 1:1 hydrochloric acid; tap water; and triple rinsed in type II reagent water.

10.3 The mass of the sample used for the analysis may require adjustment in extremely high or low calcium containing soils to achieve the required calcium concentration.

10.4 The calcium concentrations must fall between one and 4 µg/mL Ca . Samples above this range require an adjustment to the dilution in 7.2 to accomplish the required concentration. Samples below this range or samples which exhibit matrix interferences can be determined by the method of standard additions as described in US EPA, SW-846, Test Method 7000, section 8.7 .